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X-ray diffraction studies on jaggery samples

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Abstract The total sugarcane production in India is 227.06 lakh tonnes. Out of this sugarcane 43.5% is used for jaggery and khandsari [Jamale B A (2000)] The Jaggery industry is an important agro-processing industry in rural India In present paper we have undertaken X-ray diffraction studies on jaggery and thereby studied structural aspects of jaggery

Keywords Jaggery sample, X-ray diffraction.

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X-ray diffraction is the most extensively used technique to identify the crystalline phase of solid and to determine its crystal structure [1]. Here, this method has been used to identify the structure of jaggery.

A given substance always produces a characteristic diffraction pattern, whether that substance is present in the pure state or as one constituent of a mixture of substances. This fact is the basis for the diffraction method of chemical analysis. Qualitative and quantitative analysis are also possible, because the intensities of the diffraction lines due to one phase of a mixture depend on the proportion of that phase in the specimen. Detailed treatment of chemical analysis by X-ray diffraction are given by several workers [1-3].

The crystallographic features are studied by using XRD. The XRD technique based on monochromatic radiation is generally more important because the spacing of the planes (d-spacing) can be deduced from the observed diffraction angles The phenomenon of X-ray diffraction can be considered as a reflection of X-rays from the crystallographic planes of the material and is governed by Bragg's equation.

$$2d \sin\theta = n\lambda \qquad (1)$$

Where d is the lattice spacing, λ is the wavelength of monochromatic X-rays, n is the order of diffraction and θ is the diffraction angle. For thin and thick films, the powder technique in conjunction with diffractometer is most commonly used. In this instrument the diffracted radiation is detected by the counter tube, which moves along the angular range reflections. The intensities are recorded on a computer system The d values are calculated using relation (1) for known values of λ, θ and n. ? The X-rays diffraction data thus obtained is printed in tabular form on paper and compared with JCPDS (Joint committee on powder diffraction standards) data card to identify the unknown material. This X-rays diffraction data can also be used to determine dimensions of the unit cell, crystal structure and crystallinity [4, 5].

The XRD spectrum was recorded on Philips PW 3710 diffractometer attached to a digital computer alongwith graphical assembly in which CuK_α radiation source connected with the tube Cu-Ni 25kV/20 mA was used.

The X-ray diffractogram of jaggery sample records nineteen reflections between 10° and 70° (2θ) with maxima at $2\theta = 24.56°$ corresponding to a value of $d = 3.62$ A°. There is no ASTM (American Society for Testing and Materials) data available for jaggery. Hence, the indexing of the spectrogram with respect to the peaks have been carried out by using computer software and trial and error method till a good fit could be obtained

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Table 1. Powder X-ray diffraction data of jaggery sample

Peak No	2θ deg Obs	2θ deg Cal	d obs A°	d cal A°	*hkl*	RI %
1	11.51	11 51	7 682	7 682	1 2 0	20
2	12 96	12 96	6 823	6 823	2 1 0	30
3	16 54	16 54	5 354	5 354	1 0 1	20
4	18.63	18 70	4 759	4 742	3 0 1	60
5	19 37	19 27	4 579	4 601	1 2 1	60
6	19 49	19 57	4 551	4 532	2 0 1	50
7	20 16	20 19	4 399	4 395	2 1 1	20
9	21 88	21 94	4 058	4 048	2 2 1	20
10	24 56	24 60	3 622	3 616	2 3 1	100
11	25 10	25 12	3 545	3 543	0 4 1	60
12	30 76	30 76	2 904	2 904	3 5 0	20
13	31 76	31 72	2 815	2 819	2 5 1	30
15	38 33	38 32	2 346	2 347	0 7 1	40
16	39 68	39 73	2 269	2 267	5 4 1	20
17	40 16	40 19	2 243	2 242	6 1 1	20
19	50 60	50 68	1 802	1 800	1 0 1	10

between the observed and calculated 2θ and d values. The method also yielded in giving *hkl* (Miller indices) values. Comparison of the values of d and 2θ (Table 1) reveals a good agreement between the calculated and observed values of 2θ and d on the basis of assumptions of orthorhombic crystal structure, giving unit cell parameters a = 14.7459 A°, b = 17.9985 A°, C = 5.7456 and V = 1524.92 A°. In conjuction with such cell parameters the condition such as $\alpha = \beta = \gamma = 90°$ and $a \neq b \neq c$, required for the sample to be orthorhombic is found to be satisfactory [6]. Hence, it is concluded that the structure of present sample is found to be orthorhombic [7]. The grain size of the sample was determined by using Scherrers equation [8]

$$t = \frac{0.9\lambda}{B\cos\theta}$$

where, t is grain size of the sample, λ is wavelength of X-ray radiation, B is the excess of line width of radiation of the diffraction peak and θ is Bragg's angle. For the determination of grain size, the corresponding peak was enlarged for better accuracy in measuring the half width at maximum intensity.

This parameters can distinguish between natural grain size and grain size due to broadening effect which was done by calculating full width at half maximum (B) corresponding to its Bragg's angle θ and thereby computing cos and sin values.

Table 2. Data for grain size and homogeneity

Peak No.	B in radian X 10^{-4}	2θ Obs Deg	θ deg	cos θ	sin θ	Bcos θ X 10
1	1.60	11.51	5 75	0.995	0 100	1 5916
2	3.20	12.96	6.48	0 9936	0 113	3 1798
3	2 40	16.54	8.27	0 9896	0 144	2 3786
4	2.40	20 16	10 08	0 9845	0 175	2 3629
5	4.00	21 88	10 94	0 9818	0 189	3 9273
6	3 20	24 56	12 28	0.9771	0 213	3 1265
7	1 60	25 10	12 55	0.9761	0.217	1 5617
8	1 60	30 76	15.38	0.9641	0 265	1 5424
9	1 60	38 33	19 16	0 9446	0 328	
10	4.80	40 16	20 08	0 9392	0 343	4 5082
					Average =	2 9088

The nature and behavior of these values for the present sample are shown graphically in Figure 1.

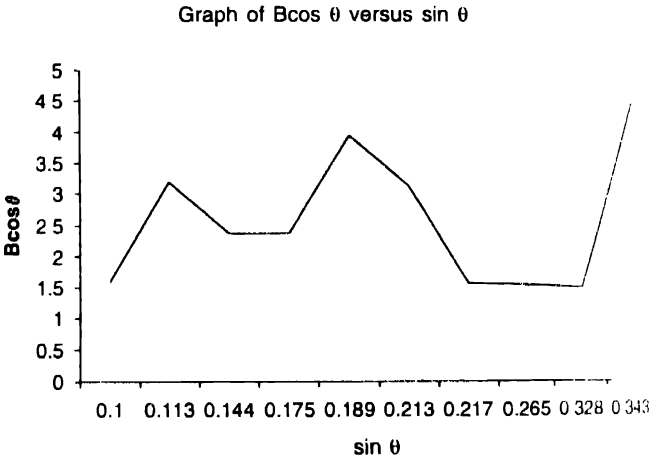

Figure 1. Graph to determine homogeneity

$$\text{Grain size } t = \frac{0.9\lambda}{B\cos\theta} = 539.756 \text{ A},$$

where λ = 1 54056 Å

It is concluded that the structure of present sample is found to be orthorhombic. A plot of $B\cos\theta$ *versus* $\sin\theta$ is not straight line indicating a presence of fluctuations. Hence, present sample seems to be non-homogeneous with respect to the particle size distortion.

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